3 MATERIALS AND METHOD

3.1 Materials and Equipment

Moringa oleifera seeds were collected from some site in Pengkalan Hulu, Perak. The chemicals are methanol, n-hexane, ferric sulphate, magnesium oxide (MgO), anhydrous sodium sulphate with analytical reagent grade. Methyl esters were produced using Soxhlet extractor fitted with a 1-L round-bottom flask, rotary evaporator, magnetic stirrer, and reflux condenser (Zhang et al., 2009).

3.2 Pre-treatment

Pre-treatment starts with collection of dry *Moringa oleifera* pods and continues with the cleaning. The dry pods were removed together with the three papery wing and light wooden shells. Cleaning process is crucial because a clean seeds yield clean oil without any impurities. After undergo cleaning process, the seeds were undergone size reduction by crushing it using mortar and pestle (Ali et al, 2010).

3.3 Oil Extraction

Approximately 20g of *Moringa oleifera* seeds are crushed and placed in a soxhlet extractor fitted with 1-L round-bottom flask and a reflux condenser. After extraction for about 1.5 hours with 250 mL of refluxing n-hexane, removal of the solvent was carried out at 50°C under vacuum using rotary evaporator to get crude *Moringa oleifera* oil which is 35% w/w.

3.4 Preparation of the Catalyst

In order to have a 1.0%, 0.7% and 0.5% (%w/w) concentration of magnesium oxide (MgO) for every 25 mL of oil used, 0.612, 0.428, and 0.306 gram of MgO was added in the methanol to produce methoxide solution. The solution was prepared in a 250 mL beaker and stirred at room temperature for about 30 minutes to activate the catalyst.

3.5 Pre-treatment

The pre-treatment was carried out to get a clean crude palm oil. In order to remove any unwanted solid particle, crude palm oil was centrifuged at 5000 rpm for 15 minutes. The clean oil was collected to proceed with tranesterification.

The experiments were carried out using the parameters shown in Table 4, and 5 for different catalyst and different methanol: oil ratios, respectively.

Table 4: Different catalyst concentration (at constant methanol:oil ratio of 12:1)

Volume of seed's oil (mL)	Catalyst concentration (%w/w)	Volume of methanol (mL)
25 (61.2 gm)	1 (0.612 gm)	300
25 (61.2 gm)	0.7 (0.428 gm)	300
25 (61.2 gm)	0.5 (0.306 gm)	300

Table 5: Different ratio of methanol: oil

Volume of seed's oil (mL)	Catalyst concentration (%w/w)	Ratio (methanol : oil)
25 (61.2 gm)	0.5 (0.306 gm)	12:1 (300:25)
25 (61.2 gm)	0.5 (0.306 gm)	10:1 (250:25)
25 (61.2 gm)	0.5 (0.306 gm)	8:1 (200:25)

3.6 Transesterification

For the transesterification process, there are two parameters to test the biodiesel production as mentioned at Table 1 and 2. For the first one, pour approximately 25 mL of *Moringa oleifera* oil into a round-bottom flask equip with a reflux condenser after the tranesterification and heat to 65°C , just below the boiling point of methanol (Zhang et al., 2009). The methanol was poured carefully into the oil. Catalyst concentration and volume of methanol was set according to Table 1. While, stirring speed, reaction time, and the reaction temperature are fixed at 200 rpm, 90 minutes, and 65°C, respectively. For the second parameters, using the same method as the first one but the best catalyst concentration and the volume of methanol was changed according to Table 2. After the

completion of the reaction, magnesium oxide (MgO) was removed by filtration and the product was left to settle overnight in a separating funnel for separation of biodiesel. The lower glycerol layer was drawn off (Zhang et al., 2009).

3.7 Washing

This method was carried out to remove glycerol using heated deionized water at 55^{o}C and poured into biodiesel. The mixture was settled in a separator funnel for 12 hours. There were 2 layer of which the lower was water + glycerol and the upper layer was biodiesel. The lower part was removed and biodiesel was continuously washed until the drain water was cleared and the pH was neutral.

3.8 Removing Excess Water

Biodiesel was heated up to 80^{o}C for 1 hours or until no more bubble on the surface of the biodiesel. The step was carried out to remove excess water from biodiesel.

3.9 Properties Determination

There are few properties that will be determined using specific methods such as cetane number. Cetane number is determined using American Standard Testing Methods D6890 (ASTM D6890). Implementing ASTM D445 and Cannon–Fenske viscometers is used to obtain the kinematic viscosity of biodiesel from *Moringa oleifera* oil. Oxidative stability measurements were carried out using a Rancimat (Metrohm, Herisau, Switzerland, equipped with software for statistical evaluation) and employing the standard EN14112. Cloud and pour point determinations were conducted with a Phase Technology (Richmond, BC, Canada) cloud, pour and freeze point analyser. Density was determined by ASTM D4052. Flash point was determined using Pensky-martens flash point – automatic NPM 440 (Normalab, France) by implementing ASTM D93 standard.

3.9.1 Cetane Number

Cetane number was determined using Ignition Quality Tester. This test was repeated three times using 50ml of biodiesel in order to get the average value and the result were tabulated. The high cetane number will help to ensure low carbon release footprint, improved fuel efficiency and reduce tear and wear for both vehicle starter and batteries (Masina et al, 2012).